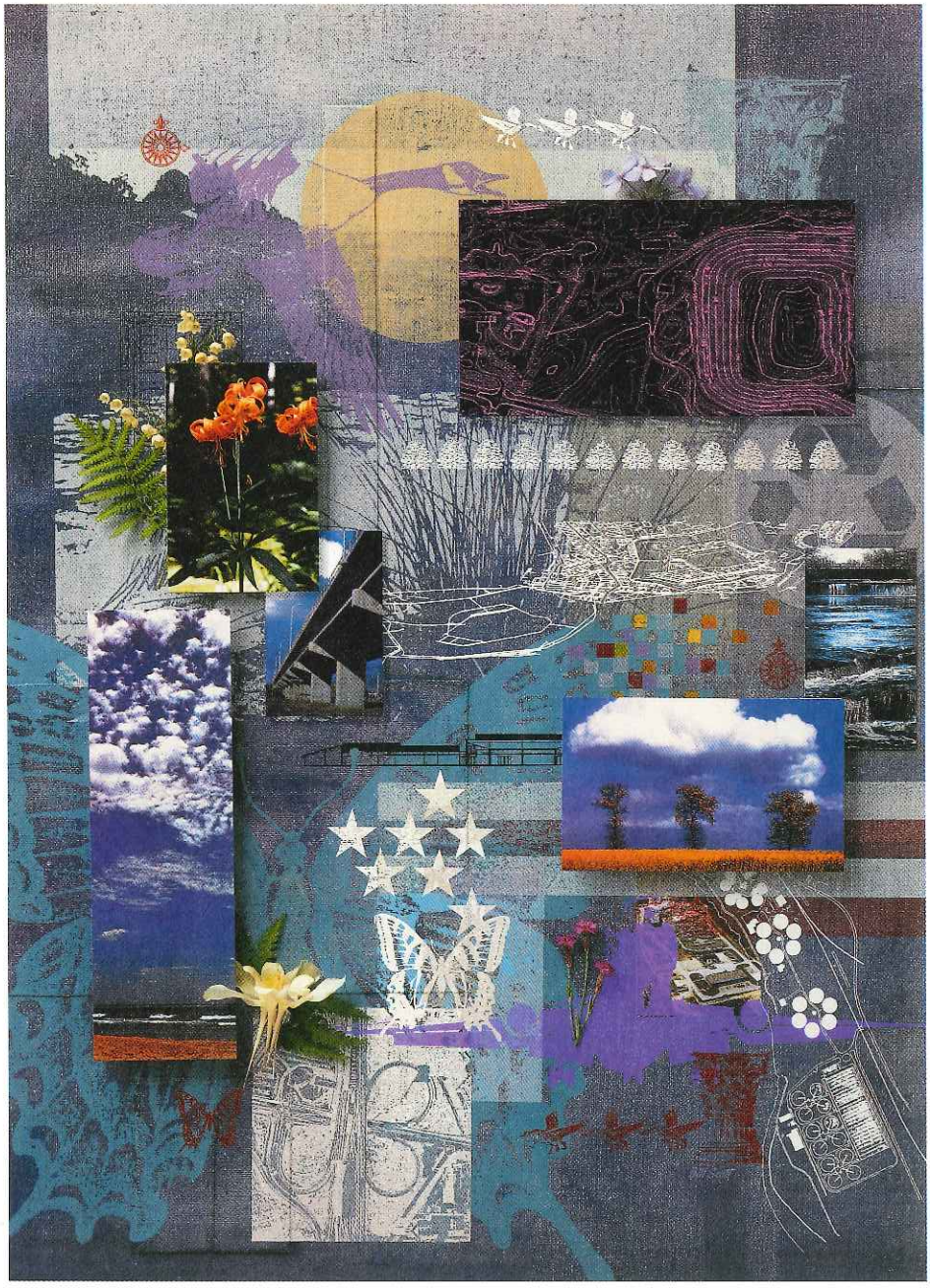
Quality • Integrity • Creativity • Responsiveness



Quality through teamwork

RCRA Facility Investigation (RFI) Phase I Work Plan

Quality Assurance Project Plan (QAPP)

Chemical Waste Management
Vickery, Ohio

U.S. EPA ID No.
OHD 020 273 819

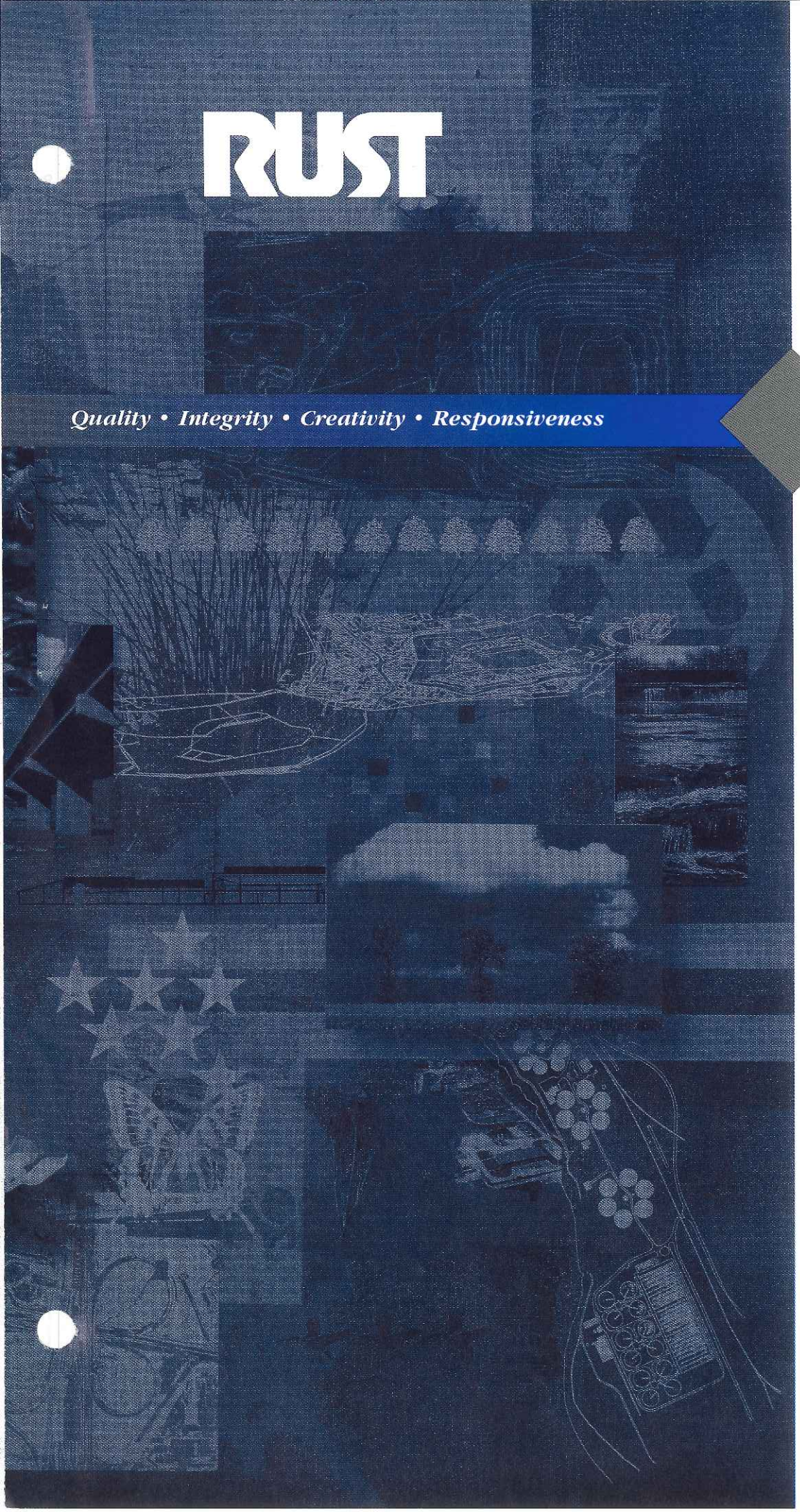
Prepared for:

CWM - Vickery, Inc.

Revised October 1998

RUST

Rust Environment & Infrastructure



RCRA Facility Investigation (RFI) Phase I Workplan

Quality Assurance Project Plan (QAPP)

Chemical Waste Management
Vickery, Ohio

U.S. EPA ID No.
OHD 020 273 819

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Revised October 1998

Rust Environment & Infrastructure

LIST OF APPENDICES

Appendix

A Laboratory Analytical Standard Operating Procedures

GP Environmental Services Standard Operating Procedures

- Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil - Method 5035, SOP No. 13.42
- Volatile Organics - Method 8260B, SOP No. 13.6
- Soil Extraction for Semi-Volatile Organics - Method 3550, SOP No. 12.17
- GC/MS Analysis of the Target Compound List and Appendix IX Semi-Volatile Organics - Method SW8270B, SOP No. 13.19
- Continuous Liquid-Liquid Extraction for Pesticide/PCBs - Method 3520B, SOP No. 12.15
- Soil Extraction for Pesticides/PCBs Compounds - Method 3550A, SOP No. 12.16
- Organochlorine Pesticides and PCBs - Method 8081, SOP No. 13.38
- Organophosphorus Pesticides - Method 8141A, SOP No. 13.36
- Analysis of Herbicides - Method 8150B, SOP No. 13.35
- Acid Digestion of Surface and Groundwater Samples for Flame/ICP Analysis and Furnace Analysis of Antimony in Accordance with SW846 - Method 3005A, SOP No. 11.36
- Acid Digestion of Soil, Sludge, Sediment and Other Solid Waste Samples for Flame/ICP and Furnace Analyses in Accordance with SW846 - Method 3050A, SOP No. 11.35
- Cold Vapor Analysis for Mercury in Accordance with SW846 - Methods 7470A and 7471A, SOP No. 11.39
- Method for Total Cyanide in Water and Soil - Method 9010A, SOP No. 11.46
- Analysis for Water and Soils for Sulfide According to MCAWW -0 Method 376.1, SOP No. 11.47
- Trace ICP Quantitation of HSL Metals plus Boron, Molybdenum, Silicon, Strontium, Titanium and Tin. SOP No. 11.62
- Chloride - MCAWW Method 300.0 - Determination of Inorganic Anions in Water and Aqueous Extract Samples by Ion Chromatography, SOP No. 11.48
- Cation Exchange Capacity of Soils, SOP 11.90

Triangle Laboratories Standard Operating Procedures

- Extraction of PCDD/PCDF from Solid Samples - 8280 and DFL M01.1, SOP No. DSP224, Version 8
- Acid/Base Cleanup of PCDD and PCDF Extracts - Method 8280, SOP No. DSP225, Version 8
- PCDDs and PCDFs by GC/MS Method 8280, SOP No. DHR183, Version 2

SECTION 1
PROJECT DESCRIPTION

This section describes the purpose and scope of the Phase I Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) at the Chemical Waste Management (CWM) Vickery Treatment, Storage and Disposal (TSD) Facility. It indicates how the findings of this investigation will be used to determine the appropriate course of further action, if any, required to address the overall objectives of the RCRA Corrective Action process. The Phase I RFI is being completed under the direction of the United States Environmental Protection Agency (U.S. EPA) as a condition to the issuance of a Part B Permit to CWM-Vickery dated October 24, 1994. As stated in the Part B Permit, CWM-Vickery must investigate potential releases of hazardous waste(s) or hazardous constituent(s) from all Solid Waste Management Units (SWMUs) at the facility, regardless of when the waste was placed in such units. If these investigations show the presence of hazardous waste constituents in the environment at concentrations that pose a risk to human health and the environment, Corrective Measures may be necessary.

This Quality Assurance Project Plan (QAPjP) presents the organization, objectives, planned activities, and specific Quality Assurance/Quality Control (QA/QC) procedures associated with the Phase I RFI for this facility. Specific protocols for sampling, sample handling and storage, chain-of-custody (COC), and laboratory and field analyses are described herein. All QA/QC procedures will be structured in accordance with applicable technical standards, U.S. EPA's requirements, regulations, guidance, and technical standards. This QAPjP has been prepared in accordance with a guidance manual entitled, "Region 5 Model RCRA Quality Assurance Project Plan," May 1993.

1.1 Introduction

This Phase I QAPjP was prepared by Rust Environment & Infrastructure (Rust) for the CWM-Vickery TSD Facility at the request of CWM-Vickery. The QAPjP is appended to the Phase I RFI Work Plan, dated August 1995, revised May 1996, November 1997, and August 1998. A Field Sampling Plan (FSP) which is included as Section 5 of the Phase I Work Plan (WP) is hereby explicitly incorporated into this QAPjP by reference. The Health and Safety Plan (HASP) is also appended to the WP.

- Additional soil or sediment sampling to further define the extent and concentration levels of hazardous waste constituents in these media;

- Sampling of existing wells where such monitoring wells are appropriately positioned to assess a particular SWMU or AOC with soil contamination; and

- Installation of additional monitoring wells where the existing monitoring well network can not adequately characterize potential groundwater impacts from a SWMU or AOC with soil contamination.

A technical memorandum, presenting the Phase I RFI data and recommendations of the QRA will be prepared and submitted to the U.S. EPA. After a review of the technical memorandum, the need for implementing a Phase II RFI will be evaluated in light of the overall objectives of the Corrective Action process.

This QAPjP is intended to fully address the scope of the Phase I RFI. To the extent that the potential Phase II RFI is comparable to the Phase I RFI, this QAPjP is expected to apply; however, specific review of any proposed Phase II RFI activities and this QAPjP will be required to assess its applicability and the need to provide further information in a "Phase II RFI QAPjP Addendum."

1.1.3 QAPjP Preparation Guidelines and History

As explained above, this Phase I RFI QAPjP has been prepared in accordance with the "Region 5 Model RCRA Quality Assurance Project Plan", dated May 1993. A pre-QAPjP meeting was held in Chicago on January 17, 1995. CWM-Vickery submitted a document describing a proposed grouping of SWMUs and outline the project objective on February 17, 1995, and received comments from U.S. EPA on April 11, 1995. The timing of this receipt did not allow the comments to be incorporated in the first draft of this QAPjP submitted in April 1995. "Version 0" of the QAPjP (August 1995) was revised in response to the February 27, 1996 comments received on "Version 0." Comments addressed during the March 25, 1996 meeting were incorporated into this QAPjP for Revision 1, dated May 1996. Subsequent comments and conditional approval of the WP and QAPjP were received in a September 11, 1997 letter from the U.S. EPA; the comments were addressed in Revision 2 of the QAPjP. On July 21, 1998, the

U.S. EPA issued a letter that required revisions to the WP and QAPjP for VOC analyses in soils. Therefore, the current version of the Phase I RFI QAPjP addresses these revisions and will be referenced as "Revision 3".

1.2 Site/Facility Description

1.2.1 Location, Facility Size, and Surroundings

As described in Section 2.1 of the Phase I RFI WP, the CWM-Vickery TSD Facility is located in a rural, unincorporated area of Sandusky County in the north-central part of Ohio.

1.2.2 Natural & Manmade Features and Topography

The natural and manmade features of the CWM-Vickery TSD Facility are described in Sections 2.1 and 3.1 of the Phase I RFI WP.

1.2.3 Local Geology & Hydrogeology

Information concerning the site's geology, soil, groundwater resources, surface hydrology and drainage is presented in Sections 3.2 and 3.3 of the Phase I RFI WP.

1.3 Site/Facility History

1.3.1 General History

The CWM-Vickery TSD Facility provides for the treatment, storage and disposal of liquid hazardous wastes. The site began as Don's Oil Service which started operations at this facility in 1958 and in 1970 changed its name to Ohio Liquid Disposal (OLD). Waste Management, Inc. (WMI) acquired the facility in 1978 and later transferred it to CWM (a wholly owned subsidiary of WMI).

The facility began as an oil recovery facility to provide hauling services for waste oil from neighboring industries to a central facility and to recover the oil for eventual resale. As time went on, the facility started to accept various industrial wastes and stored them in surface impoundments. In 1964, the facility was granted permission by the State of Ohio to accept chemical process waste. More surface impoundments were constructed to hold these wastes. As the inventory of wastes increased the site began searching for a suitable means to dispose of the waste. In 1972, OLD was granted permission to drill a test hole to evaluate the subsurface

1.6 Project Schedule

If final approval of the WP and QAPjP is obtained by September 1, 1998, field activities can begin in October 1998. Final approval of the WP and QAPjP after this date likely will result in a delay of the start date for field activities until Spring 1999. A Task Bar Chart for project activities as a function of time for September 1, 1998 approval is presented in Figure 1-1A. Figure 1-1B presents a similar schedule that includes commencement of field activities in March 1999.

project is shown in Table 1-1. Precision control limits for laboratory analysis are given in Tables 3-1 through 3-7.

3.2 Accuracy

Accuracy is the degree of agreement between an observed value and an accepted reference value. The accuracy of field measurements (HNu screening for VOCs) will be assessed through evaluation of calibration records maintained during the course of the field operations. The procedures for routine calibration of the HNu are described in Section 6 of this Phase I RFI QAPjP. Thus, no formalized assessment (i.e., the calculation of percent recovery) is planned.

Analytical accuracy will be assessed through the calculation of the percent recovery (%R) of a known amount of selected analytes spiked into a designated sample. The equation for calculating accuracy in this project can be found in Section 12 of this Phase I QAPjP. Field effects on accuracy will be assessed through evaluation of sampling documentation showing adherence with all sample handling, preservation and holding times requirements. Laboratory effects on accuracy will be assessed through evaluation of surrogate recoveries in each ORGANIC sample analyzed, and through the analysis of MS for the determination of percent recoveries. The total number of MS samples being collected for this project is shown in Table 1-1. Accuracy control limits for laboratory analysis are given in Tables 3-1 through 3-7.

For VOC analyses, MS samples will contain all of the analytes listed in Table 1-2.

3.3 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. The calculation of completeness is described in Section 12 of this QAPjP. By their nature, the field screening measurements of VOCs are considered to be 100 percent complete. It is expected that GP Environmental Services will provide data meeting QC acceptance criteria for 80 percent or

TABLE 1-2
REPORTING LIMITS AND DATA QUALITY LEVELS
APPENDIX IX VOLATILE ORGANIC COMPOUNDS BY METHOD 8260B
RFI PHASE I QAPjP
CWM-VICKERY

PARAMETERS	PROJECT REPORTING LIMIT		DATA QUALITY LEVEL (DQL)	
	SOIL (µg/kg)	WATER (µg/L)	SOIL (µg/kg)	GROUNDWATER (µg/L)
Acetone	5	10	2,000,000	610
Acetonitrile	100	100	390,000	220
Acrolein	50	20	1,300,000	730
Acrylonitrile	50	20	1,330	3.7
Allyl Chloride	10	5	3,300,000	1800
Benzene	5	0.5	1,400	0.39
Bromodichloromethane	5	5	1,400	0.18
Bromoform	5	5	56,000	8.5
Bromomethane	10	2	15,000	8.7
2-Butanone (MEK)	5	10	N/A	1900
Carbon disulfide	5	5	16,000	21
Carbon tetrachloride	5	5	470	0.17
Chlorobenzene	5	5	160,000	39
2-Chloro-1,3-butadiene (chloroprene)	5	10	N/A	N/A
Chloroethane	10	10	1,100,000	710
Chloroform	5	1	530	0.16
Chloromethane (methyl chloride)	5	10	2,000	1.5
Dibromochloromethane	5	5	5,300	1
1,2-Dibromo-3-chloropropane	10	1	320	0.048
1,2-Dibromoethane	5	1	5	0.00076
Dibromomethane	5	5	650,000	370
trans-1,4-Dichloro-2-butene	5	100	8	0.0012
Dichlorodifluoromethane	10	5	110,000	390
1,1-Dichloroethane	5	5	840,000	810
1,2-Dichloroethane	5	1	440	0.12
1,1-Dichloroethene	5	5	38	0.046
1,2-Dichloroethene (trans)	5	5	170,000	120
1,2-Dichloropropane	5	1	680	0.16
cis-1,3-Dichloropropene	5	1	510	0.081
trans-1,3-Dichloropropene	5	1	510	0.081
1,4-Dioxane	100	20	14,000	1
Ethylbenzene	5	5	2,900,000	1300
Ethyl methacrylate	5	5	340,000	550
2-Hexanone	5	10	N/A	N/A
Isobutyl alcohol	100	50	20,000,000	11000
Methacrylonitrile	100	5	1,300	1
Methylene chloride	5	1	11,000	4.3
Methyl iodide	5	5	N/A	N/A
Methyl methacrylate	5	5	5,200,000	2900
4-Methyl-2-pentanone (MIBK)	5	10	5,200,000	2900
Pentachloroethane	5	5	N/A	N/A
Propionitrile	50	50	N/A	N/A

TABLE 1-2
REPORTING LIMITS AND DATA QUALITY LEVELS
APPENDIX IX VOLATILE ORGANIC COMPOUNDS BY METHOD 8260B
RFI PHASE I QAPjP
CWM-VICKERY

PARAMETERS	PROJECT REPORTING LIMIT		DATA QUALITY LEVEL (DQL)	
	SOIL (µg/kg)	WATER (µg/L)	SOIL (µg/kg)	GROUNDWATER (µg/L)
Styrene	5	1	2,200,000	1600
1,1,1,2-Tetrachloroethane	5	1	4,800	0.43
1,1,2,2-Tetrachloroethane	5	5	900	0.055
Tetrachloroethene	5	5	7,000	1.1
Toluene	5	5	1,900,000	720
1,1,1-Trichloroethane	5	5	3,200,000	1300
1,1,2-Trichloroethane	5	1	1,400	2
Trichloroethene	5	1	7,100	1.6
Trichlorofluoromethane	10	5	710,000	1300
1,2,3-Trichloropropane	5	5	7	31
Vinyl acetate	5	10	65,000,000	37000
Vinyl chloride	10	2	5	0.02
Xylenes (total)	5	5	980,000	1400
1,4-Dichlorobenzene	5	1	7,400	0.47

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Notes:

Soil samples will be prepared in accordance with Method 5035 and GP SOP No. 13.42.

VOC Matrix Spike samples will contain all of the analytes listed above.

µg - microgram

kg - kilogram

L - Liter

N/A - DQL Not Available

SECTION 6
CALIBRATION PROCEDURES AND FREQUENCY

This section describes the calibration procedures and the frequency at which these procedures will be performed for both field and laboratory instruments.

6.1 Field Instrument Calibration

The HNu Model PI 101 (or similar instrument) will be calibrated daily prior to use and will be recalibrated every 25 samples. In addition, the PID will be calibrated if the probe assembly is removed from the readout assembly, if the ion chamber is cleaned, if the ultra-violet (UV) light is changed, or if the PID appears to be working erratically.

All calibration procedures will be documented in the field logbook. Each entry shall include the following information:

- Date;
- Time;
- Weather Conditions;
- Calibrating Personnel;
- Condition of Battery;
- Type of Calibration Gas;
- Concentration of Calibration Gas;
- Gas Cylinder Pressure;
- Span Setting; and,
- Pertinent Comments.

The PID is calibrated in the following manner:

TABLE 1-2
REPORTING LIMITS AND DATA QUALITY LEVELS
APPENDIX IX VOLATILE ORGANIC COMPOUNDS BY METHOD 8260B
RFI PHASE I QAPjP
CWM-VICKERY

PARAMETERS	PROJECT REPORTING LIMIT		DATA QUALITY LEVEL (DQL)	
	SOIL (µg/kg)	WATER (µg/L)	SOIL (µg/kg)	GROUNDWATER (µg/L)
Acetone	5	10	2,000,000	610
Acetonitrile	100	100	390,000	220
Acrolein	50	20	1,300,000	730
Acrylonitrile	50	20	1,330	3.7
Allyl Chloride	10	5	3,300,000	1800
Benzene	5	0.5	1,400	0.39
Bromodichloromethane	5	5	1,400	0.18
Bromoform	5	5	56,000	8.5
Bromomethane	10	2	15,000	8.7
2-Butanone (MEK)	5	10	N/A	1900
Carbon disulfide	5	5	16,000	21
Carbon tetrachloride	5	5	470	0.17
Chlorobenzene	5	5	160,000	39
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Chloroethane	10	10	1,100,000	710
Chloroform	5	1	530	0.16
Chloromethane (methyl chloride)	5	10	2,000	1.5
Dibromochloromethane	5	5	5,300	1
1,2-Dibromo-3-chloropropane	10	1	320	0.048
1,2-Dibromoethane	5	1	5	0.00076
Dibromomethane	5	5	650,000	370
trans-1,4-Dichloro-2-butene	5	100	8	0.0012
Dichlorodifluoromethane	10	5	110,000	390
1,1-Dichloroethane	5	5	840,000	810
1,2-Dichloroethane	5	1	440	0.12
1,1-Dichloroethene	5	5	38	0.046
1,2-Dichloroethene (trans)	5	5	170,000	120
1,2-Dichloropropane	5	1	680	0.16
cis-1,3-Dichloropropene	5	1	510	0.081
trans-1,3-Dichloropropene	5	1	510	0.081
1,4-Dioxane	100	20	14,000	1
Ethylbenzene	5	5	2,900,000	1300
Ethyl methacrylate	5	5	340,000	550
2-Hexanone	5	10	N/A	N/A
Isobutyl alcohol	100	50	20,000,000	11000
Methacrylonitrile	100	5	1,300	1
Methylene chloride	5	1	11,000	4.3
Methyl iodide	5	5	N/A	N/A
Methyl methacrylate	5	5	5,200,000	2900
4-Methyl-2-pentanone (MIBK)	5	10	5,200,000	2900
Pentachloroethane	5	5	N/A	N/A
Propionitrile	50	50	N/A	N/A

TABLE 1-2
REPORTING LIMITS AND DATA QUALITY LEVELS
APPENDIX IX VOLATILE ORGANIC COMPOUNDS BY METHOD 8260B
RFI PHASE I QAPjP
CWM-VICKERY

PARAMETERS	PROJECT REPORTING LIMIT		DATA QUALITY LEVEL (DQL)	
	SOIL (µg/kg)	WATER (µg/L)	SOIL (µg/kg)	GROUNDWATER (µg/L)
Styrene	5	1	2,200,000	1600
1,1,1,2-Tetrachloroethane	5	1	4,800	0.43
1,1,2,2-Tetrachloroethane	5	5	900	0.055
Tetrachloroethene	5	5	7,000	1.1
Toluene	5	5	1,900,000	720
1,1,1-Trichloroethane	5	5	3,200,000	1300
1,1,2-Trichloroethane	5	1	1,400	2
Trichloroethene	5	1	7,100	1.6
Trichlorofluoromethane	10	5	710,000	1300
1,2,3-Trichloropropane	5	5	7	31
Vinyl acetate	5	10	65,000,000	37000
Vinyl chloride	10	2	5	0.02
Xylenes (total)	5	5	980,000	1400
1,4-Dichlorobenzene	5	1	7,400	0.47

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Notes:

Soil samples will be prepared in accordance with Method 5035 and GP SOP No. 13.42.

µg - microgram

kg - kilogram

L - Liter

N/A - DQL Not Available

TABLE 1-3
REPORTING LIMITS AND DATA QUALITY LEVELS
APPENDIX IX SEMI-VOLATILE ORGANIC COMPOUNDS BY METHOD 8270B
RFI PHASE I QAPjP
CWM-VICKERY

PARAMETERS	PROJECT REPORTING LIMIT		DATA QUALITY LEVEL (DQL)	
	SOIL (µg/kg)	WATER (µg/L)	SOIL (µg/kg)	GROUNDWATER (µg/L)
Acenapthene	330	10	360,000	370
Acenaphthylene	330	10	N/A	N/A
Acetophenone	330	10	5,600,000	3,700
2-Acetylaminoflourene	660	20	N/A	N/A
4-Aminobiphenyl	660	20	N/A	N/A
Aniline	330	10	19,000	11
Anthracene	330	10	19,000	1,800
Aramite	165	5	18,000	2.7
Benzo(a)anthracene	330	2 1	610	0.092
Benzo(b)flouranthene	99	3	610	0.092
Benzo(k)flouranthene	99	3	6,100	0.92
Benzo(ghi)perylene	330	3	N/A	N/A
Benzo(a)pyrene	150	3	61	0.0092
Benzyl alcohol	330	10	20,000,000	11,000
bis(2-Chloroethoxy)methane	330	10	N/A	N/A
bis(2-Chloroethyl)ether	330	10	74	0.0098
bis(2-Chloroisopropyl)ether	330	10	6,300	0.96
bis(2-ethylhexyl)phthalate	330	3	32,000	4.8
4-Bromophenyl phenyl ether	330	10	N/A	N/A
Butyl benzyl phthalate	330	10	13,000,000	7,300
4-Chloroaniline	330	10	260,000	150
Chlorobenzilate	660	4	1,600	0.25
4-Chloro-3-methylphenol	330	10	N/A	N/A
2-Chloronapthalene	330	10	5,200,000	2,900
2-Chlorophenol	330	10	330,000	180
4-Chlorophenyl phenyl ether	330	10	N/A	N/A
Chrysene	330	3 1	24,000	9.2
cis/trans-Diallate	330	10	7,300	1.1
Dibenzofuran	330	10	260,000	150
Di-n-butyl phthalate	330	10	N/A	3,700
Dibenz(a,h)anthracene	150	3	61	0.0092
1,2-Dichlorobenzene	330	10	2,300,000	370
1,3-Dichlorobenzene	330	10	2,800,000	N/A

TABLE 1-3
REPORTING LIMITS AND DATA QUALITY LEVELS
APPENDIX IX SEMI-VOLATILE ORGANIC COMPOUNDS BY METHOD 8270B
RFI PHASE I QAPjP
CWM-VICKERY

PARAMETERS	PROJECT REPORTING LIMIT		DATA QUALITY LEVEL (DQL)	
	SOIL (µg/kg)	WATER (µg/L)	SOIL (µg/kg)	GROUNDWATER (µg/L)
3,3'-Dichlorobenzidine	660	20	990	0.15
2,4-Dichlorophenol	330	10	200,000	110
2,6-Dichlorophenol	330	10	N/A	N/A
Diethyl phthalate	330	10	52,000,000	29,000
Dimethoate	99	3	13,000	7.3
p-(Dimethylamino)azobenzene	330	10	N/A	N/A
7,12-Dimethylbenz(a)anthracene	330	10	N/A	N/A
3,3'-Dimethylbenzidine	330	10	48	0.0073
a,a-Dimethylphenethylamine	330	10	N/A	N/A
2,4-Dimethylphenol	330	10	1,300,000	730
Dimethyl phthalate	330	10	>100000000	370,000
1,3-Dinotrobenzene	0	3	6,500	3.7
4,6-Dinitro-2-methylphenol	1,650	50	N/A	N/A
2,4-Dinitrophenol	1,650	50	130,000	73
2,4-Dinitrotoluene	330	10	130,000	73
2,6-Dinitrotoluene	330	10	65,000	37
Di-n-octyl phthalate	330	10	1,300,000	730
Diphenylamine	330	10	1,600,000	910
Ethyl methanesulfonate	660	20	N/A	N/A
Fluoranthene	330	10	2,600,000	1,500
Fluorene	330	10	300,000	240
Hexachlorobenzene	200	3	280	0.042
Hexachlorobutadiene	330	3	5,700	0.86
Hexachlorocyclopentadiene	330	10	450,000	260
Hexachloroethane	330	10	32,000	4.8
Hexachloropropene	330	10	N/A	N/A
Indeno(1,2,3-cd)pyrene	330	2	610	0.092
Isodrin	660	20	N/A	N/A
Isophorone	330	10	470,000	71
Isosafrole	660	10	N/A	N/A
Methapyrilene	3,300	100	N/A	N/A
3-Methylcholanthrene	330	10	N/A	N/A
Methyl methansulfonate	495	15	N/A	N/A

TABLE 1-3
REPORTING LIMITS AND DATA QUALITY LEVELS
APPENDIX IX SEMI-VOLATILE ORGANIC COMPOUNDS BY METHOD 8270B
RFI PHASE I QAPjP
CWM-VICKERY

PARAMETERS	PROJECT REPORTING LIMIT		DATA QUALITY LEVEL (DQL)	
	SOIL (µg/kg)	WATER (µg/L)	SOIL (µg/kg)	GROUNDWATER (µg/L)
2-Methylnaphthalene	330	10	N/A	N/A
4-Methylphenol	330	10	330,000	180
2 or 3-Methylphenol	330	10	3,300,000	1,800
Naphthalene	330	10	800,000	240
1,4-Naphthoquinone	330	10	N/A	N/A
1-Naphthalamine	330	10	N/A	N/A
2-Naphthalamine	330	10	N/A	N/A
2-Nitroanaline	1,650	10	3,900	2.2
3-Nitroanaline	1,650	50	N/A	N/A
4-Nitroanaline	660	20	N/A	N/A
Nitrobenzene	330	10	33,000	18
2-Nitrophenol	330	10	N/A	N/A
4-Nitrophenol	1,650	50	N/A	N/A
4-Nitroquinoline-1-oxide	1,320	40	N/A	N/A
n-Nitrosodi-n-butylamine	330	10	82	0.012
n-Nitrosodiethylamine	660	20	3	0.00045
n-Nitrosodimethylamine	165	5	9	0.0013
n-Nitrosodiphenylamine	330	10	91,000	14
n-Nitrosodi-n-propylamine	165	5	63	0.0096
n-Nitroso-n-methylethylamine	330	10	20	0.0031
n-Nitrosomorpholine	330	10	N/A	N/A
n-Nitrosopiperidine	660	20	N/A	N/A
n-Nitrosopyrrolidine	330	10	210	0.032
5-Nitro-o-toluidine	330	10	N/A	N/A
Pentachlorobenzene	330	10	52,000	29
Pentachloronitrobenzene	660	20	1,700	0.26
Pentachlorophenol	330	10	2,500	0.56
Phenacetin	660	20	N/A	N/A
Phenanthrene	330	10	N/A	N/A
Phenol	330	10	39,000,000	22,000
1,4-Phenylenediamine	330	10	12,000,000	6,900
2-Picoline	330	10	N/A	N/A
Pronamide	330	10	4,900,000	2,700

TABLE 1-3
REPORTING LIMITS AND DATA QUALITY LEVELS
APPENDIX IX SEMI-VOLATILE ORGANIC COMPOUNDS BY METHOD 8270B
RFI PHASE I QAPjP
CWM-VICKERY

PARAMETERS	PROJECT REPORTING LIMIT		DATA QUALITY LEVEL (DQL)	
	SOIL (µg/kg)	WATER (µg/L)	SOIL (µg/kg)	GROUNDWATER (µg/L)
Pyrene	330	10	2,000,000	1,100
Pyridine	330	10	65,000	37
Safrole	330	10	N/A	N/A
1,2,4,5-Tetrachlorobenzene	330	10	20,000	11
2,3,4,6-Tetrachlorophenol	330	10	2,000,000	1,100
Tetraethyl dithiopyrophosphate	330	10	33,000	18
Thionazine	660	20	N/A	N/A
o-Toluidine	330	10	N/A	N/A
1,2,4-Trichlorobenzene	330	10	620,000	190
2,4,5-Trichlorophenol	330	10	6,500,000	3,700
2,4,6-Trichlorophenol	0	5	40,000	6.1
o,o,o-Triethyl phosphorothioate	330	10	N/A	N/A
1,3,5-Trinitrobenzene	0	2	3,300	1.8

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Notes:

µg - microgram
kg - kilogram
L- Liter
N/A - DQL Not Available

TABLE 1-6
REPORTING LIMITS AND DATA QUALITY LEVELS
APPENDIX IX ORGANOCHLORINE HERBICIDES BY METHOD 8150B
RFI PHASE I QAPjP
CWM-VICKERY

PARAMETERS	PROJECT REPORTING LIMIT		DATA QUALITY LEVEL (DQL)	
	SOIL (µg/kg)	WATER (µg/L)	SOIL (µg/kg)	GROUNDWATER (µg/L)
2,4-D	500	1.0	650,000	370
Dinoseb	100	0.7	65,000	37
2,4,5-TP (Silvex)	100	0.5	520,000	290
2,4,5-T	250	0.5	650,000	370

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Notes:

2,4-D - 2,4 - Dichlorophenoxyacetic Acid

2,4,5 - TP (Silvex) 2-(2,4,5 -Trichlorophenoxy) Propionic Acid

2,4,5 - T - 2,4,5 - Trichlorophenoxyacetic Acid

µg - microgram

kg - kilogram

L - Liter

TABLE 1-7
REPORTING LIMITS AND DATA QUALITY LEVELS
APPENDIX IX METALS AND INORGANICS
RFI PHASE I QAPjP
CWM-VICKERY

PARAMETERS	PROJECT REPORTING LIMIT		DATA QUALITY LEVEL (DQL)		ANALYTICAL METHOD
	SOIL (µg/kg)	WATER (µg/L)	SOIL (µg/kg)	GROUNDWATER (µg/L)	
Antimony	170	1.7	31,000	15	7041
Antimony	500	5.0	31,000	15	6010A
Arsenic	300	3.0	320	0.038	6010A
Barium	1000	10.0	5,300,000	2,600	6010A
Beryllium	100	1.0	140	0.016	6010A
Cadmium	500	5.0	38,000	18	6010A
Chromium	500	5.0	210,000	180	6010A
Cobalt	1000	10.0	N/A	N/A	6010A
Copper	1000	10.0	2,800,000	1,400	6010A
Lead	300	3	400,000	4	6010A
Mercury	200	0.2	23,000	11	7471A
Nickel	2000	20.0	1,500,000	730	6010A
Selenium	500	5	380,000	180	6010A
Silver	300	3	380,000	180	6010A
Thallium	1000	10	6,100	2.9	6010A
Tin	1000	10.0	46,000,000	22,000	6010A
Vanadium	500	5	540,000	260	6010A
Zinc	500	5.0	23,000,000	11,000	6010A
Chloride	25,000	N/A	N/A	N/A	300.0
Cyanide	1000	5.0	380,000	6.2	9010A
Sulfide	5000	1,000.0	N/A	N/A	376.1

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Notes:

*** - The Graphite Frunance Atomic Absorption methods will be employed as a backup to the Inductively Coupled Plasma method**
µg - microgram
kg - kilogram
L - Liter
N/A - Not Applicable

TABLE 3-1
PRECISION AND ACCURACY CRITERIA
APPENDIX IX VOLATILE ORGANIC COMPOUNDS BY METHOD 8260B
RFI PHASE I QAPjP
CWM-VICKERY

PARAMETER	PRECISION (RPD)		ACCURACY (%R)	
	Water	Soil	Water	Soil
PARAMETER	PRECISION (RPD)		ACCURACY (%R)	
	Water	Soil	Water	Soil
Benzene	11	21	76-127	66-142
Chlorobenzene	13	21	75-130	60-133
1,1-Dichloroethene	14	22	61-145	59-172
Toluene	13	21	76-125	59-139
Trichloroethene	14	24	71-120	62-137

SURROGATE COMPOUND	PRECISION (RPD)		ACCURACY (%R)	
	Water	Soil	Water	Soil
4 Bromofluorobenzene	N/A	N/A	86-115	74-121
1, 2 DichloroEthane - d4	N/A	N/A	80-120	70-121
Toluene - d8	N/A	N/A	88-110	81-117

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Note:
Soil samples will be prepared in accordance with Method 5035 and GP SOP No. 13.42.
RPD - Relative Percent Difference
%R - Percent Recovery
N/A - Not Applicable

TABLE 4-2
SAMPLE CONTAINERS, PRESERVATIVES and HOLDING TIMES
RFI PHASE I QAPjP
CWM-VICKERY

Parameter	Media[1]	Container	Preservation	Maximum Holding Time[2]
Volatile Organics	Water[6]	(2) 40 mi VOA Vials with Teflon© lined septum	4 drops HCL, Cool to 4°C	14 days
	Soil	(3) En-Core™ samplers	Cool to 4°C	2 days/14 days[7]
Semi-Volatiles, Pesticides, PCBs, Herbicides	Water[6]	(5) 1 L amber glass with Teflon© liner	Cool to 4°C	47 days[1]
	Soil	(2) 4 oz. wide mouth glass with Teflon© liner	Cool to 4°C	54 days[4]
Metals[5]	Water	1 L polyethylene with polyethylene liner	50% HNO_3, pH <2. Cool to 4°C	6 months
Metals, Cyanide, Sulfide, Chloride	Soil	(1) 4 oz. wide mouth glass with Teflon© liner	Cool to 4°C	6 months
Cyanide	Water	1 L polyethylene with polyethylene liner	NaOH. pH > 12. Cool to 4°C	14 days
Sulfide	Water	1 L polyethylene with polyethylene liner	Zinc Acetate, Cool to 4°C	7 days
PCDD, PCDF	Soil	(1) 4 oz. wide mouth glass with Teflon© liner	Cool to 4°C	54 days[4]
	Water[6]	1 L narrow-mouth amber glass with Teflon© liner	Cool to 4°C	47 days[3]
Geotechnical Parameters	Soil	(1) 16 oz. wide mouth glass with Teflon© liner	Cool to 4°C	45 days

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Notes:

1. Water refers to subsurface water or surface water samples. Soil refers to samples from test pits, soil borings and streambed sediments.
2. Holding time refers to the number of days following the Validated Time of Sample collection by the laboratory.
3. Seven days until extraction and analysis within 40 days of extraction.
4. Fourteen days until extraction and analysis within 40 days of extraction. For PCDD/PCDF, 30 days until extraction and analysis within 40 days of extraction.
5. Mercury has a holding time of 28 days for both liquid and soil samples. Liquid samples will be preserved to pH <2 with 50% nitric acid and cooled to 4°C. Soil samples will be cooled to 4°C.
6. Matrix Spike/Matrix Spike Duplicate will require three times this sample volume.
7. 2 days for preparation, 14 days for analysis.

L - liter
c - centigrade
oz - ounce
PCDD - Polychlorinated Dibenzo-p-Dioxins
PCDF - Polychlorinated Dibenzofurans
NaOH - Sodium Hydroxide
HCL - Hydrochloric Acid
HNO_3 - Nitric Acid

TABLE 7-1
ANALYTICAL & PREPARATORY METHODS
RFI PHASE I QAPjP
CWM-VICKERY

GP Environmental Services SOP No.	Title	3rd Edition SW 846 Method No.
13.42	Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples	5035
13.6	Volatile Organics	8260B
12.17	Soil Extraction for Semivolatile Organics (Sonication Extraction)	3550A
13.19	GC/MS Analysis of The Target Compound List and Appendix IX Semivolatile Organics	8270B
12.15	Continuous Liquid-Liquid Extraction for Pesticides/PCBs	3520B
12.16	Soil Extraction for Pesticides/PCBs Compounds (Sonication Extraction)	3550A
13.38	Organochlorine Pesticides and PCBs	8081
13.36	Organophosphorus Pesticides	8141A
13.35	Analysis of Herbicides	8150B
11.36	Acid Digestion of Surface and Ground Water Samples for Flame/ICP Analyses and Furnace Analysis of Antimony in Accordance with SW 846	3005A
11.35	Acid Digestion of Soil, Sludge, Sediment, and Other Solid Waste Samples for Flame/ICP and Furnace Analyses in Accordance with SW 846	3050A
11.62	Trace ICP Quantitation of HSL Metals	6010A
11.39	Cold Vapor Analysis for Mercury in Accordance with SW 846	7470A & 7471A
11.46	Method for Total Cyanide Analysis In Water and Soil	9010A
11.47	Analysis for Water and Soils for Sulfide According to MCAWW	376.1
11.48	Inorganic Anions	300.0
Triangle No. DSP224	Extraction of PCDD/PCDF from Solid Samples - 8280 and DFL M01.1	8280
Triangle No. DSP225	Acid/Base Cleanup of PCDD and PCDF Extracts - Method 8280	8280
Triangle No. DSP183	PCDDs and PCDFs by GC/MS Method 8280	8280

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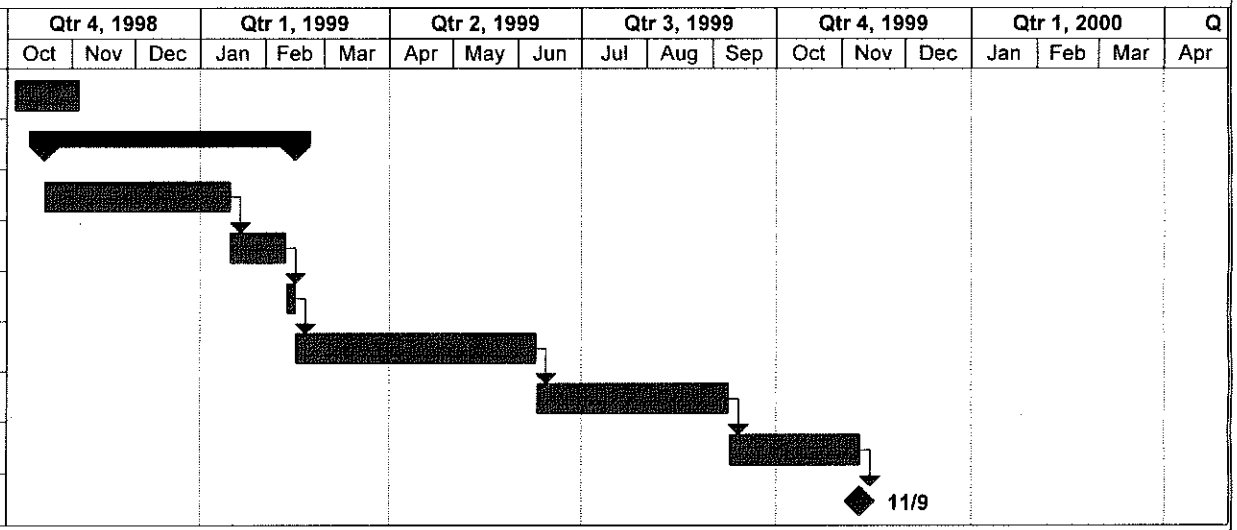
Note:
GC/MS - Gas Chromatography/Mass Spectrometry
PCBs - Polychlorinated Biphenyls
ICP - Inductively Coupled Plasma
*** - The Graphite Furnance Atomic Absorption methods will be employed as a backup to the ICP methods**
HSL - Hazardous Substance List
MCAWW - Method for the Chemical Analysis of Waste and Water

Figure 1-1A
RFI Phase I Schedule of Activities
CWM-Vickery

ID	Task Name	Start	Duration	Finish
1	**Mobilization**	10/5/98	18d	11/4/98
2	**Field Investigation**	**10/19/98**	**62d**	**2/15/99**
3	SWMU Groups A through I	10/19/98	45d	1/15/99
4	Areas of Concern	1/15/99	15d	2/10/99
5	Groundwater Sampling	2/11/99	2d	2/15/99
6	**RFI Final Report**	2/15/99	66d	6/9/99
7	**EPA Review**	6/10/99	52d	9/8/99
8	**Address Comments**	9/9/99	35d	11/9/99
9	**Submit Final Report**	11/9/99	0d	11/9/99



Project: CWM-Vickery RFI Work Plan
Date: 8/24/98

Task
Progress
Milestone
Summary
Rolled Up Task
Rolled Up Milestone
Rolled Up Progress

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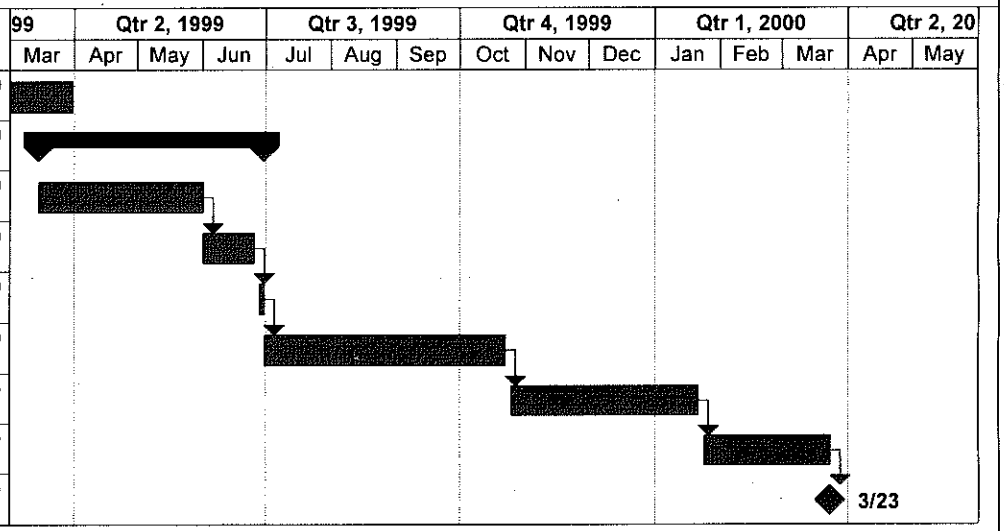
Figure 1-1B
RFI Phase I Schedule of Activities
CWM-Vickery

ID	Task Name	Start	Duration	Finish
1	**Mobilization**	3/1/99	18d	3/31/99
2	**Field Investigation**	**3/15/99**	**62d**	**6/30/99**
3	SWMU Groups A through I	3/15/99	45d	6/1/99
4	Areas of Concern	6/1/99	15d	6/25/99
5	Groundwater Sampling	6/28/99	2d	6/30/99
6	**RFI Final Report**	6/30/99	66d	10/22/99
7	**EPA Review**	10/25/99	52d	1/21/00
8	**Address Comments**	1/24/00	35d	3/23/00
9	**Submit Final Report**	3/23/00	0d	3/23/00



Project: CWM-Vickery RFI Work Plan
Date: 8/24/98

Task
Progress
Milestone
Summary
Rolled Up Task
Rolled Up Milestone
Rolled Up Progress

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SOP No: 13.42

Title: Method 5035 – Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples

1.0 SCOPE AND APPLICATION

1.1 This method describes a closed-system purge-and-trap process for the analysis of volatile organic compounds (VOCs) in solid materials (e.g., soils, sediments, and solid waste). While the method is designed for use on samples containing low levels of VOCs, procedures are also provided for collecting and preparing solid samples containing high concentrations of VOCs and for oily wastes. These procedures may be used in conjunction with any appropriate determinative gas chromatographic procedure, including, but not limited to, Methods 8015; 8021, and 8260.

2.0 SUMMARY OF METHOD

2.1 Low concentration soil method – generally applicable to and soils and other solid samples with VOC concentrations in the range of 0.5 to 200µg/kg.

Volatile organic compounds (VOCs) are determined by collecting an approximately 5g sample, weighed in the field at the time of collection, and placing it in a pre-weighed vial with a septum-sealed screw-cap (see Sec. 4) that already contains a stirring bar and a sodium bisulfate preservative solution. The vial is sealed and shipped to the laboratory. The entire vial is then placed, unopened, into the instrument carousel. Immediately before analysis, organic-free reagent water, surrogates, and internal standards (if applicable) are automatically added without opening the sample vial. The vial containing the sample is heated to 40°C and the volatiles purged into an appropriate trap using an inert gas combined with agitation of the sample. Purged components travel via a transfer line to a trap. When purging is complete, the trap is heated and backflushed with helium to desorb the trapped sample components into a gas chromatograph for analysis by an appropriate determinative method.

2.2 High concentration soil method – generally applicable to soils and other solid samples with VOC concentrations greater than 200µg/kg.

The sample introduction technique in Sec. 2.1 is not applicable to all samples, particularly those containing high concentrations (generally greater than 200µg/kg) of VOCs which may overload either the volatile trapping material or exceed the working range of the determinative instrument system (e.g., GC/MS, GC/FID, GC/EC, etc.). In such instances, this method describes two sample collection options and the corresponding sample purging procedures.

2.2.1 The first option is to collect a bulk sample in a vial or other suitable container without the use of the preservative solution described in Sec. 2.1. A portion of that sample is removed from the container in the laboratory and is dispersed in a water-miscible solvent to dissolve the volatile organic constituents. An aliquot of the solution is added to 5mL of reagent water in a purge tube. Surrogates and internal standards (if applicable) are added to the solution, then purged using Method 5030, and analyzed by an appropriate determinative method. Because the procedure involves opening the vial and removing a portion of the soil, some volatile constituents may be lost during handling.

2.2.2 The second option is to collect an approximately 5g sample in a pre-weighed vial with a septum-sealed screw-cap that contains 5mL of a water-miscible organic solvent (e.g., methanol). At the time of analysis, surrogates are added to the vial, then an aliquot of the solvent is removed from the vial, purged using Method 5030 and analyzed by an appropriate determinative method.

2.3 High concentration oily waste method – generally applicable to oily samples with VOC concentrations greater than 200µg/kg that can be diluted in a water-miscible solvent.

3.0 INTERFERENCES

3.1 Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running method blanks. The use of non-polytetrafluoroethylene (non-PTFE) plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging device must be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. These compounds will result in interferences or false positives in the determinative step.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and acetone) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free reagent water and carried through sampling and handling protocols serves as a check on such contamination.

3.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. Where practical, samples with unusually high concentrations of analytes should be followed by an analysis of organic-free reagent water to check for cross-contamination. If the target compounds present in an unusually concentrated sample are also found to be present in the subsequent samples, the analyst must demonstrate that the compounds are not due to carryover. Conversely, if those target compounds are not present in the subsequent sample, then the analysis of organic-free reagent water is not necessary.

3.4 The laboratory where volatile analysis is performed should be completely free of solvents. Special precautions must be taken to determine methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory workers' clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination. The presence of other organic solvents in the laboratory where volatile organics are analyzed will also lead to random background levels and the same precautions must be taken.

4.0 APPARATUS AND MATERIALS

4.1 Sample Containers

The purge and trap system employed at GPES require 40ml clear screw cap vials with PTFE lined low bleed septa.

4.2 Purge-and-Trap System

The purge-and-trap system consists of a Tekmar LSC-2000 concentrator equipped with an Archon autosampler that automatically adds water, surrogates, and internal standards (if applicable) to a vial containing the sample, purges the VOCs using an inert gas stream while agitating the contents of the vial, and also traps the released VOCs for subsequent desorption into the gas chromatograph.

4.2.1 The purging device is capable of accepting 40ml vial that contains a 5g soil sample plus a magnetic stirring bar and 10mL of water. The device is capable of heating a soil vial to 40°C and

holding it at that temperature while the inert purge gas is allowed to pass through the sample. The device is also capable of introducing at least 5mL of organic-free reagent water into the sample vial while trapping the displaced headspace vapors. It is also capable of agitating the sealed sample during purging (using a magnetic stirring bar added to the vial prior to sample collection). The analytes being purged is quantitatively transferred to an absorber trap.

4.2.1.1 The trap used for this method is 25cm long, with an inside diameter of 0.105 inches, made of charcoal and marketed under VOCARB 3000.

4.2.2 The desorber for the trap must be capable of rapidly heating the trap to the temperature recommended by the trap material manufacturer, prior to the beginning of the flow of desorption gas. Several commercial desorbers (purge-and-trap units) are available.

4.3 Syringe and Syringe Valves

4.3.1 25mL glass hypodermic syringes with Luer-Lok (or equivalent) tip (other sizes are acceptable depending on sample volume used).

4.3.2 2-way syringe valves with Luer ends.

4.3.3 25µL micro syringe with a 2 inch x 0.006 inch ID, 22° bevel needle (Hamilton #702N or equivalent).

4.3.4 Micro syringes – 10, 100µL.

4.3.5 Syringes – 0.5, 1.0, and 5mL, gas-tight with shut-off valve.

4.4 Miscellaneous

4.4.1 Glass vials

4.4.1.1 40mL, screw-cap, PTFE lined, septum-sealed. Examine each vial prior to use to ensure that the vial has a flat, uniform sealing surface.

4.4.2 Top-loading balance – Capable of accurately weighing to 0.01g.

4.4.3 Glass scintillation vials – 20mL, with screw-caps and PTFE liners, or glass culture tubes with screw-caps and PTFE liners, for dilution of oily waste samples.

4.4.4 Volumetric flasks – Class A, 10mL and 100mL, with ground-glass stoppers.

4.4.5 2mL glass vials, for GC autosampler – used for oily waste samples extracted with methanol of PEG.

4.4.6 Spatula, stainless steel – narrow enough to fit into a sample vial.

4.4.7 Disposable Pasteur pipettes.

4.4.8 Magnetic stirring bars – PTFE-coated. Stirring bars are reused, provided that they are thoroughly cleaned between uses.

5.0 REAGENTS

5.1 Organic-free reagent water – all references to water in this method refer to organic-free reagent water, as defined in Chapter One of SW846.

5.2 Methanol, CH_3OH – purge-and-trap quality or equivalent. Store away from other solvents.

5.3 Low concentration sample preservative.

5.3.1 Sodium bisulfate, $NaHSO_4$ – ACS reagent grade or equivalent.

5.3.2 The preservative should be added to the vial prior to shipment to the field, and must be present in the vial prior to adding the sample.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Preparation of sample vials

The specific preparation procedures for sample vials depend on the expected concentration range of the sample, with separate preparation procedures for low concentration soil samples and high concentration soil and solid waste samples. Sample vials should be prepared in a fixed laboratory or other controlled environment, sealed, and shipped to the field location. Gloves should be worn during the preparation steps.

6.1.1. Low concentration soil samples

The following steps apply to the preparation of vials used in the collection of low concentration soil samples to be analyzed by the closed-system purge-and-trap equipment described in this SOP.

6.1.1.1 Add a clean magnetic stirring bar to each clean vial.

6.1.1.2 Add preservative to each vial. The preservative is added to each vial prior to shipping the vial to the field. Add approximately 1g of sodium bisulfate to each vial. If samples markedly smaller or larger than 5g are to be

collected, adjust the amount of preservative added to correspond to approximately 0.2g of preservative for each 1g of sample. Enough sodium bisulfate should be present to ensure a sample pH of ≤2.

6.1.1.3 Add 5mL of organic-free reagent water to each vial. The water and the preservative will form an acid solution that will reduce or eliminate the majority of the biological activity in the sample, thereby preventing biodegradation of the volatile target analytes. If an efferescing matrix is encountered samples should be handled according to the procedure note in Section 6.2.1.2.

6.1.1.4 Seal the vial with the screw-cap and septum seal.

6.1.1.5 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible).

6.1.1.6 Weigh the prepared vial to the nearest 0.01g, record the tare weight, and write it on the label.

6.1.1.7 Because volatile organics will partition into the headspace of the vial from the aqueous solution and will be lost when the vial is opened, surrogates, matrix spikes, and internal standards (if applicable) should only be added to the vials after the sample has been added to the vial. These standards should be introduced back in the laboratory automatically by the sample introduction system, just prior to analysis.

6.1.2. High concentration soil samples collected without a preservative.

When high concentration samples are collected without a preservative, a variety of sample containers may be employed, including 60mL glass vials with septum seals (see Sec. 4.4).

6.1.3 High concentration soil samples collected and preserved in the field.

The following steps apply to the preparation of vials used in the collection of high concentration soil samples to be preserved in the field with methanol and analyzed by the aqueous purge-and-trap equipment described in Method 5030.

6.1.3.1 Add 10mL of methanol to each vial.

6.1.3.2 Seal the vial with the screw-cap and septum seal.

6.1.3.3 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible).

6.1.3.4 Weigh the prepared vial to the nearest 0.01g, record the weight, and write it on the label. Vials containing methanol should be weighed a second time on the day that they are to be used. Vials found to have lost methanol (reduction in weight of >0.01g) should not be used for sample collection.

6.1.3.5 Surrogates, internal standards and matrix spikes (if applicable) should be added to the vial containing the soil sample after it is returned to the laboratory and prior to analysis.

6.2 Sample Collection

This section is not applicable to the laboratory operation, however, familiarity with sample collection would help the analyst with proper implementation of Method 5035.

Collect the sample according to the procedures outlined in the sampling plan. As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of the volatile components. Several techniques may be used to transfer a sample to the relatively narrow opening of the low concentration soil vial. These include devices such as the EnCore™ sampler, the Purge-and-Trap Soil Sampler™, and a cut plastic syringe. Always wear gloves whenever handling the tared sample vials.

6.2.1 Low concentration soil samples

6.2.1.1 Using an appropriate sample collection device, collect approximately 5g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere: generally within a few minutes at most. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.

6.2.1.2 Using the sample collection device, add about 5g (2-3cm) of soil to the sample vial containing the preservative solution. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap. Store samples on ice at 4°C.

NOTE: Soil samples that contain carbonate minerals (either from natural sources or applied as an amendment) may effervesce upon contact with the acidic preservative solution in the low concentration sample vial. If the amount of gas generated is very small (i.e., several mL), any loss of volatiles as a result of such effervescence may be minimal if the vial is sealed quickly. However, if larger amounts of gas are generated, not only may the sample lose a significant amount of analyte, but the gas pressure may shatter the vial if the sample vial is sealed. Therefore, when samples are known or suspected to contain high levels of carbonates, a test sample should be collected, added to a vial, and a checked for effervescence. If a rapid or vigorous reaction occurs, discard the sample and collect low concentration samples in vials that do not contain the preservative solution. Add 5ml of reagent grade organic free water to the sample vial. Then samples should be shipped to the lab within 48 hours of collection while kept at 4° ± 2°. Upon sample receipt, the lab must freeze the samples at temperatures below minus 10°C for up to a period of 12 days. Overall sample analysis should be performed within 14 days from the collection date. Sample vials must be kept tilted in the freezer to minimize the circumferential pressure of water expansion on the glass vial.

6.2.1.3 When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 ± 0.5g of sample were added. The balance should be calibrated in the field using an appropriate weight for the sample containers employed (Sec. 4.5.5). Record the weight of the sealed vial containing the sample to the nearest 0.01g.

6.2.1.4 Alternatively, collect several trial samples with plastic syringes. Weigh each trial sample and note the length of the soil column in the syringe. Use these data to determine the length of soil in the syringe that corresponds to 5.0 ± 0.5g. Discard each trial sample.

6.2.1.5 As with the collection of aqueous samples for volatiles, collect at least two replicate samples. This will allow the laboratory an additional sample for re-analysis. The second sample should be taken from the same soil stratum or the same section of the solid waste being sampled, and within close proximity to the location from which the original sample was collected.

6.2.1.6 In addition, since the soil vial cannot be opened without compromising the integrity of the sample, at least one additional aliquot of sample must be collected for screening, dry weight determination, and high concentration analysis (if necessary). This third aliquot may be collected in a 60mL glass vial or a third 40mL soil sample vial. However, this third vial must not contain the sample preservative solution, as an aliquot will be used to determine dry weight. If high concentration samples are collected in vials containing methanol, then two additional aliquots should be collected, one for high concentration analysis collected in a vial containing methanol, and another for the dry weight determination in a vial without either methanol or the low concentration aqueous preservative solution.

6.2.1.7 If samples are known or expected to contain target analytes over a wide range of concentrations, thereby requiring the analyses of multiple sample aliquots, it may be advisable and practical to take an additional sample aliquot in a low concentration soil vial containing the preservative, but collecting only 1-2g instead of the 5g collected in Sec. 6.2.1.1. This aliquot may be used for those analytes that exceed the instrument calibration range in the 5g analysis.

6.2.1.8 The EnCore™ sampler has not been thoroughly evaluated by EPA as a sample storage device. While preliminary results indicate that storage in the EnCore™ device may be appropriate for up to 48 hours, samples collected in this device should be transferred to the soil sample vials as soon as possible, or analyzed within 48 hours. Samples should be taken in triplicates (per site) and shipped to the lab immediately after collection. The lab should transfer the EnCore™ cartridges to clean 40ml vials and add aliquots of water to two vials and methanol to the third one (follow the procedure in Section 6).

6.2.1.9 The collection of low concentration soil samples in vials that contain methanol is not appropriate for samples analyzed with the closed-system purge-and-trap equipment described in this method (see Sec. 6.2.2).

6.2.2 High concentration soil samples preserved in the field.

The collection of soil samples in vials that contain methanol has been suggested by some as a combined preservation and extraction procedure. However, this procedure is not appropriate for use with the low concentration soil procedure described in this method.

6.2.2.1 When samples are known to contain volatiles at concentrations high enough that the dilution factor will not preclude obtaining results within the calibration range of the appropriate determinative method, a sample may be collected and immediately placed in a sample vial containing purge-and-trap grade methanol.

6.2.2.2 Using an appropriate sample collection device, collect approximately 5g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere: generally within a few minutes at most. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.

6.2.2.3 Using the sample collection device, add about 5g (2-3cm) of soil to the vial containing 10mL of methanol. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap. Store samples on ice at 4°C.

6.2.2.4 When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 ± 0.5g of sample were added. The balance should be calibrated in the field using an appropriate weight for the sample containers employed (Sec. 4.5.5). Record the weight of the sealed vial containing the sample to the nearest 0.01g.

6.2.2.5 Alternatively, collect several trial samples with plastic syringes. Weigh each trial sample and note the length of the soil column in the syringe. Use these data to determine the length of soil in the syringe that corresponds to 5.0 ± 0.5g. Discard each trial sample.

6.2.2.6 Other sample weights and volumes of methanol may be employed, provided that the analyst can demonstrate that the sensitivity of the overall analytical procedure is appropriate for the intended application.

6.2.2.7 The collection of at least one additional sample aliquot is required for the determination of the dry weight, as described in Sec. 6.2.1.6. Samples collected in methanol should be shipped as described in Sec. 6.3, and must be clearly labeled as containing methanol, so that the samples are not analyzed using the closed-system purge-and-trap equipment described in this procedure.

6.2.3 High concentration soil sample not preserved in the field.

The collection of high concentration soil samples that are not preserved in the field generally follows similar procedures as for the other types of samples described in Secs. 6.2.1 and 6.2.2, with the obvious exception that the sample vials contain neither the aqueous preservative solution nor methanol. However, when field preservation is not employed, it is better to collect a larger volume sample, filling the sample container as full as practical in order to minimize the headspace. Such collection procedures generally do not require the collection of a separate aliquot for dry weight determination, but it may be advisable to collect a second sample aliquot for screening purposes, in order to minimize the loss of volatiles in either aliquot.

6.3 Sample handling and shipment

All samples for volatiles analysis should be cooled to approximately 4°C, packed in appropriate containers, and shipped to the laboratory on ice, as described in the sampling plan.

6.4 Sample Storage

6.4.1 Once in the laboratory, store samples at 4°C until analysis. The sample storage area should be free of organic solvent vapors.

6.4.2 All samples should be analyzed within 14 days from collection date.

6.4.3 Sample collected in EnCore™ cartridges should be analyzed within 48 hours from collection or transferred to 40ml VOA vials, preserved and analyzed within 12 days. Refer to Section 6.2.1.2.

6.4.4 When the low concentration samples are strongly alkaline or highly calcareous in nature, the sodium bisulfate preservative solution may not be strong enough to reduce the pH of the soil/water solution to below 2. Therefore, when low concentration soils to be sampled are known or suspected to be strongly alkaline or highly calcareous, additional steps may be required to preserve the samples. Such steps include: addition of larger amounts of the sodium bisulfate preservation to non-calcareous samples, storage of low concentration samples at -10°C (taking care not to fill the vials so full that the expansion of the water in the vial breaks the vial), or significantly reducing the maximum holding time for low concentration soil samples. Whichever steps are employed, they should be clearly described in the sampling and QA project plans and distributed to both the field and laboratory personnel. See Sec. 6.2.1.2 for additional information.

7.0 PROCEDURE

This section describes procedures for the low concentration soil method and the high concentration soil method. High concentration samples are to be introduced into the purge-and-trap system using Method 5030.

7.1 Low concentration soil method (approximate concentration range of 0.5 to 200µg/kg – the concentration range is dependent upon the determinative method and the sensitivity of each analyte).

7.1.1 Initial Calibration

Prior to using this introduction technique for any GC or GC/MS method, the system must be calibrated. General calibration procedures are discussed in the related GC or GCMS method SOPs. Normally, external standard calibration is preferred for the GC methods (non-MS detection) because of possible interference problems with internal standards. If interferences are not a problem, or when a GC/MS method is used, internal standard calibration may be employed.

7.1.1.1 Assemble a purge-and-trap device that meets the specification in Sec. 4.2 and that is connected to a gas chromatograph or a gas chromatograph/mass spectrometer system.

7.1.1.2 Before initial use, a VOCARB 3000 trap should be conditioned 2 hours at 245°C by backflushing with an inert gas flow of at least 20mL/minute. Prior to daily use, the trap should be conditioned for 10 minutes at 245°C with backflushing.

7.1.1.3 Establish the purge-and-trap instrument operating conditions. Adjust the instrument to inject 5mL of water, to heat the sample to 40°C, and to hold the sample at 40°C for 1.5 minutes before commencing the purge process, or as recommended by the instrument manufacturer.

7.1.1.4 Prepare a minimum of five initial calibration standards containing all the analytes of interest and surrogates, as described in the Method SOP, and following the instrument manufacturer's instructions. The calibration standards are prepared in organic-free reagent water. The volume of organic-free reagent water used for calibration must be the same as the volume used for sample analysis (5mL added to the vial before shipping it to the field plus the organic-free reagent water added by the instrument). The calibration standards should also

contain approximately the same amount of the sodium bisulfate preservative as the sample (e.g., ~1g), as the presence of the preservative will affect the purging efficiencies of the analytes. The internal standard solution must be added automatically, by the instrument, in the same fashion as used for the samples. Place the soil vial containing the solution in the instrument carousel. In order to calibrate the surrogates using standards at five concentrations, it is necessary to disable the automatic addition of surrogates to each vial containing a calibration standard. Prior to purging, heat the sample vial to 40°C for 1.5 minutes.

7.1.2 Continuing Calibration

Refer to Method SOPs for details on continuing calibration. A single standard near the mid-point of calibration range is used for verification. This standard should also contain approximately 1g of sodium bisulfate.

7.1.3 Sample purge-and-trap

This method is designed for a 5g sample size, but smaller sample sizes may be used. The soil vial is hermetically sealed at the sampling site, and MUST remain so in order to guarantee the integrity of the sample. Gloves must be worn when handling the sample vial since the vial has been tared. If any soil is noted on the exterior of the vial or cap, it must be carefully removed prior to weighing. Weigh the vial and contents to the nearest 0.01g, even if the sample weight was determined in the field, and record this weight. This second weighing provides a check on the field sampling procedures and provide additional assurance that the reported sample weight is accurate. Data users should be advised on significant discrepancies between the field and laboratory weights.

7.1.3.1 Remove the sample vial from storage and allow it to warm to room temperature. Shake the vial gently, to ensure that the contents move freely and that stirring will be effective. Place the sample vial in the instrument carousel.

7.1.3.2 Without disturbing the hermetic seal on the sample vial, add 5mL of organic-free reagent water, the internal standards, and the surrogate compounds. This is carried out using the automated sampler. Other volumes of organic-free reagent water may be used, however, it is imperative that all samples, blanks, and calibration standards have exactly the same final volume of organic-

free reagent water. Prior to purging, heat the sample vial to 40°C for 1.5 minutes.

7.1.3.3 For the sample selected for matrix spiking, add the matrix spiking solution described I Sec. 5.0 of Method 5000, either manually, or automatically. The concentration of the spiking solution and the amount added should be established as described in the related SOP.

7.1.3.4 Purge the sample with helium at a flow rate of up to 40mL/minute (the flow rate may vary from 20 to 40mL/min, depending on the target analyte group) for 11 minutes while the sample is being agitated with the magnetic stirring bar or other mechanical means. The purged analytes are allowed to flow out of the vial through a glass-lined transfer line to a trap.

7.1.4 Sample Desorption

After the 11 minute purge, place the purge-and-trap system in the desorb mode and preheat the trap to 245°C without a flow of desorption gas. Start the flow of desorption gas at 10mL/minute for about two minutes. Begin the temperature program of the gas chromatograph and start data acquisition.

7.1.5 Trap Reconditioning

After desorbing the sample for 4 minutes, recondition the trap by returning the purge-and-trap system to the purge mode. Maintain the trap temperature at 245°C. After approximately 8 minutes, turn off the trap heater and halt the purge flow through the trap. When the trap is cool, the next sample can be analyzed.

7.1.6 Data Interpretation

Perform qualitative and quantitative analysis following the guidance given in the determinative methods and Method SOPs. If the concentration of any target analyte exceeds the calibration range of the instrument, it will be necessary to reanalyze the sample by the high concentration method. Such re-analyses need only address those analytes for which the concentration exceeded the calibration range of the low concentration method. Alternatively, if a sample aliquot of 1-2g was also collected (see Sec. 6.2.1.7), it may be practical to analyze that aliquot for the analytes that exceeded the instrument calibration range in the 5g analysis.

7.2 High concentration method for soil samples with concentrations generally greater than 200µg/kg.

The high concentration method for soil is based on a solvent extraction. A solid sample is either extracted or diluted, depending on sample solubility in a water-miscible solvent. An aliquot of the extract is added to organic-free reagent water containing surrogates and, if applicable, internal and matrix spiking standards, purged according to Method 5030, and analyzed by an appropriate determinative method.

The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared using the steps below, beginning at Sec. 7.2.1. If solvent preservation was employed in the field, then the preparation begins with Sec. 7.2.4.

7.2.1 When the high concentration sample is not preserved in the field, the sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Whenever practical, mix the contents of the sample container by shaking or other mechanical means without opening the vial. When shaking is not practical, quickly mix the contents of the vial with a narrow metal spatula and immediately reseal the vial.

7.2.2 For soil and solid waste samples that are soluble in methanol, add 9.0mL of methanol and 1.0mL of the surrogate spiking solution to a tared 20mL vial. Using a top-loading balance, weigh 5g (wet weight) of sample into the vial. Quickly cap the vial and reweigh the vial. Record the weight to 0.1g. Shake the vial for 2 min. If the sample was not soluble in methanol, but was soluble in PEG, employ the same procedure described above, but use 9.0mL of PEG in place of the methanol. Proceed with Sec. 7.2.5.

7.2.3 For soil and solid waste samples that were collected in methanol or PEG (see Sec. 6.2.2), weigh the vial to 0.1g as a check on the weight recorded in the field, add the surrogate spiking solution to the vial by injecting it through the septum, shake for 2 min, as described above, and proceed with Sec. 7.2.5.

7.2.4 Pipette approximately 1mL of the extract from either Sec. 7.3.3 or 7.3.4 into a GC vial for storage, using a disposable pipette, and seal the vial. The remainder of the extract may be discarded. Add approximately 1mL of methanol or PEG to a separate GC vial for use as the method blank for each set of samples extracted with the same solvent.

7.2.5 The extracts must be stored at 4°C in the dark, prior to analysis. Add an appropriate aliquot of the extract (see Table 2) to 5.0mL of organic-free reagent water and analyze by Method 5030 in conjunction with the appropriate determinative method.

7.2.6 If results are to be reported on a dry weight basis, determine the dry weight of a separate aliquot of the sample, using the procedure in Sec. 7.3, after the sample extract has been transferred to a GC vial and the vial sealed.

7.3 Determination of % Dry Weight

If results are to be reported on a dry weight basis, it is necessary to determine the dry weight of the sample.

NOTE: It is highly recommended that the dry weight determination only be made after the analyst has determined that no sample aliquots will be taken from the 60mL vial for high concentration analysis. This is to minimize loss of volatiles and to avoid sample contamination from the laboratory atmosphere. There is no holding time associated with the dry weight determination. Thus, this determination can be made any time prior to reporting the sample results, as long as the vial containing the additional sample has remained sealed and properly stored.

7.3.1 Weigh 5-10g of the sample from the 60mL VOA vial into a tared crucible.

7.3.2 Dry this aliquot overnight at 105°C. Allow to cool in a desiccator before weighing. Calculate the % dry weight as follows:

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$